IS 3295-2 (1970): Method of chemical analysis of ferroboron, Part 2: Determination of boron [MTD 5: Ferro Alloys]

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REAFFIRMED

METHOD OF CHEMICAL ANALYSIS OF FERRO BORON

PART II DETERMINATION OF BORON

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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METHOD OF CHEMICAL ANALYSIS OF FERRO BORON

PART II DETERMINATION OF BORON

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TO

IS : 3295 (Part II)-1970 METHOD OF CHEMICAL ANALYSIS OF FERRO BORON

PART II DETERMINATION OF BORON

Alteration

(*Page* 5, *clause* 4.4.5) — Substitute the following for the existing clause:

'4.4.5 Neutralise with dilute sodium hydroxide solution until the colour changes to purple. Add sufficient mannitol solution so that the resulting solution contains 11 g of mannitol per 100 ml of solution. Titrate with standard sodium hydroxide solution until the colour of the solution changes to purple.'

(SMDC 2)

Reprography Unit, BIS, New Delhi, India

Indian Standard

METHOD OF CHEMICAL ANALYSIS OF FERRO BORON

PART II DETERMINATION OF BORON

0. FOREWORD

0.1 This Indian Standard (Part II) was adopted by the Indian Standards Institution on 22 June 1970, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Chemical analysis of ferro boron, chemical composition of which is specified in IS : 3013-1965*, is covered in two parts of this standard. In this part, method for determination of boron in ferro boron is covered. The methods for determination of carbon, silicon and aluminium in ferro boron are given in Part I of this standard.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

1. SCOPE

1.1 This standard (Part II) prescribes the method for determination of boron in ferro boron.

2. SAMPLING

2.1 Samples shall be drawn and prepared in accordance with the procedure laid down for the purpose of chemical analysis in IS : 1472 (Part I)-1959‡.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1960§) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Specification for ferro boron.
†Rules for rounding off numerical values (*revised*).
‡Methods of sampling ferro alloys, Part I.
§Specification for water, distilled quality (*revised*).

4. DETERMINATION OF BORON BY THE ION EXCHANGE (VOLUMETRIC) METHOD

4.1 Outline of the Method — The sample is dissolved in sulphuric acid and nitric acid mixture. Any residue left is brought into solution by fusion and acidification. The cations are removed by passing the solution through a column of an ion exchange resin. After acidification of the effluent, mannitol is added and solution titrated with standard sodium hydroxide solution.

4.2 Ion Exchange Column — Make a column (300 × 20 mm dia) from glass tubing filled with a suitable ion exchange resin (*see* Note) taking care that no air gap remains inside the column. Place a plug of glass woolin the bottom of the column. Wash the column with hydrochloric acid (6N) followed by water until the washings are neutral.

NOTE — Zeo carb 225 (H^+) has been found satisfactory for this purpose.

4.3 Reagents

4.3.1 *Concentrated Sulphuric Acid* — sp gr 1·84 (conforming to IS:266-1961*).

4.3.2 *Concentrated Nitric Acid* — sp gr 1·42 (conforming to IS:264-1968†).

4.3.3 *Dilute Hydrochloric Acid* — 1 percent (*v/v*).

4.3.4 *Fusion Mixture* — equimolecular quantities of solid anhydrous sodium carbonate and potassium carbonate.

4.3.5 *Potassium Nitrate* — solid.

4.3.6 *Mixed Indicator* — Dissolve 0·05 g of methyl red, 0·1 g of bromocresol green, 0·3 g of phenolphthalein and 0·3 g of thymolphthalein in 100 ml of methyl alcohol by digesting at 50°C.

4.3.7 *Dilute Sodium Hydroxide Solution* — 10 percent (*w/v*).

4.3.8 *Hydrochloric Acid Solution* — N/10 approximately. Dilute 9 ml of concentrated hydrochloric acid to one litre and standardize against sodium carbonate.

4.3.9 *Mannitol Solution* — 30 percent (*w/v*).

4.3.10 *Standard Sodium Hydroxide Solution* — N/10, carbonate free. Standardize with potassium biphthalate (AR).

4.4 Procedure

4.4.1 Crush the sample so that it passes through 150-micron IS Sieve and take 1 g of the sample in 250-ml round bottom flask fitted with a

*Specification for sulphuric acid (*revised*).
†Specification for nitric acid (*first revision*).

ground-glass joint. Add 75 ml of water, 5 ml of concentrated sulphuric acid and 3·5 ml of concentrated nitric acid. Fix a condenser and gently heat until the main reaction ceases. Boil the solution for 30 minutes, cool, wash down the condenser and the sides of the beaker. Filter the solution through a paper pulp pad and wash with minimum quantity of dilute hydrochloric acid.

4.4.2 Transfer the paper pulp and residue in a platinum crucible and ignite at a low temperature until carbonaceous matter is burnt off and then add 0·5 g of fusion mixture and 50 mg of potassium nitrate and heat to fusion for 10 minutes. Cool the melt and extract in 100 ml of water and add the solution to the main solution. Transfer the solution to 500-ml volumetric flask and make up to the mark and mix well.

4.4.3 Pipette out 50 ml of the solution and pour it on the top of the resin column and allow the solution to pass through the column at the rate of nearly 5 ml per minute. Wash the column with 250 ml of water, collect the effluent in a 500-ml conical flask.

4.4.4 Add 15 drops of mixed indicator to the effluent and continue to add dropwise dilute sodium hydroxide solution until the colour changes to purple. Make the solution acidic again by adding dropwise hydrochloric acid solution until the colour changes to definite red. Boil with a reflux condenser for 10 minutes and then cool.

4.4.5 Add sufficient mannitol solution so that the resulting solution contains 11 g of mannitol per 100 ml of solution. Titrate with standard sodium hydroxide solution until the colour of the solution changes to green.

4.4.6 Carry out a blank determination following the same procedure and using the same amount of all reagents, but without the sample.

4.5 Calculation

$$\text{Boron, percent} = \frac{(A - B) \times C \times 0{\cdot}1082}{D}$$

where

A = volume in ml of standard sodium hydroxide solution used for titration,

B = volume in ml of standard sodium hydroxide solution required for the blank titration,

C = normality factor of the standard sodium hydroxide solution, and

D = weight in g of the sample representing the aliquot taken.

NOTE — In case phosphorus is present, suitable correction should be made.

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